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**Progress Report for the Division
of Safeguards and Security:
July-December 1976**

June 29, 1977

Monsanto

MOUND LABORATORY

Miamisburg, Ohio
operated by

MONSANTO RESEARCH CORPORATION

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for the

**UNITED STATES ENERGY RESEARCH
AND DEVELOPMENT ADMINISTRATION**

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R. P. Ratay, Editor

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Foreword

Under the sponsorship of the ERDA Division of Safeguards and Security, Mound Laboratory is responsible for studies to advance nuclear safeguards technology in the public interest.

This report is submitted by W. T. Cave, Director, Nuclear Operations, and R. E. Vallee, Manager of Technology Applications and Development, from contributions prepared by members of the Applied Physics Section: W. H. Smith, Manager.

Summaries

DEVELOPMENT OF NONDESTRUCTIVE ASSAY MEASUREMENTS

Prediction of Calorimeter Equilibrium

A newly derived single-exponential prediction equation has been shown to yield results equal to those from previously reported work, but allows for more versatility in the instrumentation selection. A single-precision rather than a double-precision calculator is all that is required to predict reliable equilibrium values using this new equation. A double-exponential prediction equation has also been derived and tested using simulated data. (Page 5)

SYSTEMS DEVELOPMENT

Automated Plutonium Assay System

Engineering refinements have been incorporated in the Automated Plutonium Assay System (APAS) to provide for improvements in the gamma-ray spectroscopy system and the container pickup device. In addition, evaluation tests on the assay instrumentation have been conducted. Calorimeter tests indicate that values within 1% of equilibrium can be obtained in 7 min. (Page 6)

APPLICATIONS

Half-Life Study

The Half-Life Evaluation Committee has completed measurements of the half-life of plutonium-239. Preliminary results show that measurements by alpha and gamma counting, by mass spectrometry and by calorimetry agree sufficiently well that a recommended value with a relative standard deviation of +0.1% will be reported. Papers describing these measurements are being prepared for journal publication. (Page 7)

The committee concurs with the recent recommendation by Argonne National Laboratory representatives that the value 87.74 ± 0.04 yr be adopted for the half-life of plutonium-238.

NDA Reference Standards

Calorimetric assay has been used to prepare plutonium reference standards for use in verifying other NDA measurement systems. The reference samples contained plutonium oxide, nitride, and/or metal mixed with natural, depleted, and/or enriched uranium oxide, nitride, and/or metal. Eight samples have been certified, with plutonium uncertainties of less than 1%. They cover the weight range from 4.3 to 141.2 g of plutonium. (Page 10)

Development of Nondestructive Assay Measurements

Prediction of Calorimeter Equilibrium

INTRODUCTION

Prediction-of-equilibrium studies have continued using a new single-exponential prediction equation. The new equation yields results equal to those from the previous equation [1,2] and allows for more versatility in instrument selection since the calculations can be done using single precision. A double-exponential prediction equation has also been derived and tested using simulated data.

SINGLE-EXPONENTIAL MODEL

A different approach has been used to derive the single-exponential prediction equation. The original derivations [2] (Equation 1) gave a predicted value A based on the difference between a squared data point and the product of two points.

$$A = \frac{Y_2^2 - Y_1 Y_3}{2Y_2 - Y_1 - Y_3} \quad (1)$$

This new derivation (Equation 2) allows for the prediction based on simple differences between data points:

$$A = \frac{Y_2 - Y_1 \left(\frac{Y_3 - Y_2}{Y_2 - Y_1} \right)}{1 - \left(\frac{Y_3 - Y_2}{Y_2 - Y_1} \right)} \quad (2)$$

Although the two derivations are equal, the use of Equation 2 has a definite advantage. Valid prediction computations based on the difference between observed data points can be achieved using a single-precision minicomputer. However,

when using Equation 1 double precision is required to avoid rounding errors.

Numerous sample analyses have been made using this second derivation to verify previous results.

UNCERTAINTY IN THE PREDICTED VALUE

An uncertainty or accuracy level relative to the final calorimeter value should be associated with the predicted equilibrium value. It would also be useful to be able to establish an acceptable accuracy level before starting the prediction analysis, which could be a key parameter for terminating the calorimeter run. Using the basic prediction program (Equation 2), an estimate of the relative accuracy of the predicted value has been derived (Equation 3) and the parameters which control the accuracy have been identified.

$$\sigma_A \approx 1.96ue^{-\lambda\epsilon}/(1 - e^{-\lambda\epsilon})^2 \quad (3)$$

The uncertainty in the observed data points (u) and the time constant ($1/\lambda$) are fixed parameters for a given calorimeter system. The only parameter which can be varied to affect the predicted value is the interval between points (ϵ). Given a desired level of relative uncertainty in A, the optimum time interval between points can be calculated and the observation rate can be set accordingly. The standard deviation of 10-point groups of predicted values will reach a minimum equal to or less than the desired level of accuracy.

DOUBLE-EXPONENTIAL PREDICTION EQUATION

A double-exponential prediction equation (Equation 4) has been derived based on differences between five individual data points:

$$A = \frac{C_0 Y_1 + C_1 Y_2 + Y_3}{C_0 + C_1 + 1} \quad (4)$$

where:

$$C_0 = - \frac{(Y_3 - Y_4)^2 - (Y_2 - Y_3)(Y_4 - Y_5)}{(Y_3 - Y_4)(Y_1 - Y_2) - (Y_2 - Y_3)^2}$$

$$C_1 = - \frac{(Y_2 - Y_3)(Y_3 - Y_4) - (Y_1 - Y_2)(Y_4 - Y_5)}{(Y_2 - Y_3)^2 - (Y_1 - Y_2)(Y_3 - Y_4)}$$

The equation has been successfully tested on simulated data, but it has not yet been used to analyze actual calorimetric data. (C. L. Fellers and P. W. Seabaugh)

Systems Development

Automated Plutonium Assay System

INTRODUCTION

The Automated Plutonium Assay System (APAS) is being developed to evaluate and demonstrate the operating characteristics of an in-line assay system for mixed-oxide fuels [3]. The three-glove-box system consists of a three-dimensional sample transporter, two calorimeters (with provisions for four additional calorimeters), a gamma-ray spectrometer, and a central process computer which will control sample movement in the box line and collect and reduce data from the assay instruments. These processed data will provide material control and safeguards accountability.

GAMMA-RAY SPECTROSCOPY SYSTEM

In order to average out any inhomogeneities in the plutonium distribution in the sample cans, the cans will be rotated while the gamma-ray spectra are acquired. The rotator assembly consists of a synchronous motor with the planetary gear reduction.

Because of the confined space in which the motor operates, the temperature rise due to its operation had to be determined.

Two preliminary maximum-temperature tests were conducted by continuously running the motor for 8 hr.

A copper-constantan thermocouple was fastened to the motor housing; the maximum temperature observed was 52°C, which is within the operating limits of the motor. Further tests will be performed when the lead shield is installed around the gamma-ray well.

Because the rotator assembly is hidden in a sample well, a Hall-effect sensor has been designed to sense the rotation of the turntable. This sensor will be continuously monitored during the time the gamma-ray spectra are being acquired.

To ensure minimum downtime if the synchronous motor needs to be replaced, a spring-loaded hermetically sealed connector has been installed in the sample well. This connector will allow motor replacement without destroying the integrity of the glovebox line.

A positioning cart for the liquid nitrogen dewar was designed, fabricated, and installed. The cart allows the dewar to be moved into position and locked so it may be moved in only one dimension. After alignment with the collimator the cart position is fixed.

PICKUP DEVICE

The dual-state pickup device used for securing sample cans during movement from one location to another in the system has begun to show some wear on an internal bearing surface following long-term reliability tests. As a result, the device occasionally failed to either release or pick up a can. Engineering modifications have been made to increase

the pickup device reliability. A shoulder screw was incorporated to allow for improved positioning of the sleeve bearings, and the inner plunger with the machined cam was changed to 440C stainless steel and hardened for improved bearing surface (Figure 1). Reliability tests will be continued to ensure that these modifications were successful in improving the device performance.

CALORIMETRY

A series of tests was run and reported [4] on one of the two APAS calorimeters to determine the time per analysis required with and without preequilibrated samples. Figures 2, 3, and 4 show the typical response of the calorimeter as a ratio of the observed-to-equilibrium values plotted versus time for 0.5, 1, and 3-W samples, respectively. For preequilibrated samples, the calorimeter response is within 1% of the equilibrium value in 7 min. To obtain the same value on non-preequilibrated samples, 40 to 70 min is required, depending on the sample wattage. These latter times are representative of the time required to move a single sample through the calorimeter. However, if a backlog of samples exists, the use of preequilibration can be very beneficial for rapidly moving samples through the system.

SOFTWARE DEVELOPMENT

Although the software interface program for the calorimeters has been developed and successfully tested, the decision has been made to modify this program to incorporate additional instrumentation. A microprocessor will be interfaced between the calorimeters and the central process computers to facilitate the handling of large amounts of data which will

be collected from the calorimeters at equal time intervals. Calorimeter data will be stored in the microprocessor and transmitted to the computer when the computer is idle. The specifications for the microprocessor have been written and the purchase order has been placed. Program modification is currently being conducted. (J. H. Birden, A. R. Campbell, M. F. Duff, C. L. Fellers, J. F. Lemming and J. R. Wetzel)

Applications

Half-Life Study

INTRODUCTION

The half-life evaluation committee consisting of representatives from Mound Laboratory, LLL, LASL, ANL, NBS, and Rockwell (RFP) is conducting a half-life measurement program to resolve discrepancies in published values of plutonium half-lives.

The program calls for half-life measurements of plutonium isotopes to be made on aliquots from a single metal sample of high isotopic and chemical purity. Three different half-life measurement techniques (calorimetry, alpha counting, and mass spectrometry) are being used.

PLUTONIUM-239

All of the participating laboratories have completed their measurements on the plutonium-239 samples. The preliminary results were presented to the committee. It was decided not to recommend a composite value until statistical analyses are performed. The committee has decided to publish its work as a group of papers describing individual efforts with a recommended half-life presented in an introductory

FIGURE 1 - Dual-state pickup.

FIGURE 2 - Wattage ratios as a function of time for the 0.5-W sample.

FIGURE 3 - Wattage ratios as a function of time for the 1-W sample.

FIGURE 4 - Wattage ratios as a function of time for the 3-W sample.

paper. The committee will meet May 16-17, 1977, at NBS to review drafts of all papers prior to submission to a journal.

PLUTONIUM-238

The committee reviewed the measurement of the half-life of plutonium-238 by Jaffey, ANL, who observed the plutonium-238 α -particle activity formed from the α -decay of curium-242. The details of this measurement have been published [5].

Jaffey's measured value, 87.71 ± 0.03 yr, is in excellent agreement with the value of 87.77 ± 0.03 yr obtained by K. C. Jordan [6] using calorimetry. Because of this agreement with independent techniques, it was concluded that no further need existed to continue a planned effort to measure the half-life of plutonium-238.

The committee concurred with Jaffey that the average of his and Jordan's value, 87.74 ± 0.04 yr, be accepted as the best value for the half-life of plutonium-238. (W. W. Strohm)

NDA Reference Standards

INTRODUCTION

Calorimetric assay is traceable to the national measurement system. Because calorimetry is independent of the sample matrix, it can be used to determine plutonium values for cans which contain mixed matrices such as powder and pellets. In this reporting period 15 samples of varying chemical forms and physical forms have been assayed.

SAMPLES

Table 1 summarizes the chemical composition and the physical matrices of the samples. The samples are generally plutonium and uranium oxides or nitrides. The uranium-235 isotopic content varies from depleted to 93% enriched.

ISOTOPIC COMPOSITION

Three aliquots were chosen by the shipper from each of the 15 samples in order to verify the isotopic composition of the samples.

The relative isotopic abundances for the 15 samples have been determined using two gamma-ray spectroscopy systems. Similar measurements were made on the 45 aliquots. Subsequently the aliquots were analyzed by radiocounting and mass spectroscopy.

The aliquot values from each sample were combined using an unweighted average to give an estimate of the isotopic abundances of the sample. For the 45 aliquots, the plutonium isotopic abundances determined by gamma-ray spectroscopy agree with those determined by chemical analysis within the measurement uncertainties. The ratios (gamma-ray value/chemical value) for the plutonium isotopes determined from the aliquots are: plutonium-238/plutonium-239 - 0.97 ± 0.06 ; plutonium-240/plutonium-239 - 0.94 ± 0.04 ; and plutonium-241/plutonium-239 - 0.98 ± 0.04 .

For some samples, large differences between aliquots from the same sample indicate possible inhomogeneities in the sample.

Table 1 - SAMPLE PARAMETERS

Sample	Chemical Form	Physical Form
AP	PuO ₂ + UO ₂ (natural)	Powder and SS capsule
247	PuO ₂ + UO ₂ (natural)	Powder
655	Pu + U (depleted)	In metal mounts
712	PuO ₂ + UO ₂ (natural)	Powder and pellets
858	{ PuN + UN (enriched)	Pellets, powder, filings
	{ PuN + UN (depleted)	Pellets
888	PuN + UN (enriched)	Pellets
889	PuN + UN (enriched)	Pellets
901	PuO ₂ + UO ₂ (natural)	Pellets
906	PuO ₂ + UO ₂ (natural)	Pellets
914	Pu metal, PuN + UN	
	(enriched)	Pellets, oxide
930	PuO ₂ + UO ₂ (depleted)	Powder
931	PuO ₂ + UO ₂ (depleted)	Powder
945	PuO ₂ + UO ₂ (depleted)	Pellets
961	PuN + UN (enriched)	Pellets and powder
978	PuN + UN (enriched)	Powder and pellets

For two samples agreement between the three aliquots but disagreement between the sample value and the aliquots indicates sampling problems.

For 13 of the samples, the best-estimate value from the aliquots agrees with the gamma-ray determination from the bulk sample. The ratios of the plutonium isotopes are: plutonium-238/plutonium-239 - 0.98 ± 0.11 ; plutonium-240/plutonium-239 - 1.04 ± 0.06 ; and plutonium-241/plutonium-239 - 1.02 ± 0.07 . The isotopic abundances for the other two samples differ significantly from the best-estimate value of their aliquots.

Because of the sample inhomogeneities and/or sampling problems, the samples are grouped in three categories: I) those for which the aliquots do not agree with the sample; II) those for which there are large variations in the aliquots; and III) those for which there are no discrepancies.

For the first group, the can and aliquot values were averaged and the standard deviation is used for the uncertainties. For the second and third groups, the estimated value from the aliquots has been used for the plutonium isotopic ratios. For the second group, the uncertainties are determined from the larger of the standard deviation between aliquots or the standard deviation of replicate measurements on the same aliquot. The uncertainties for the third group are based on the average standard deviation for that group.

The americium-241 value has been determined by averaging the gamma-ray value

and radiocounting value on the aliquots. The uncertainty used for the americium-241 value is 10% except in those cases where the standard deviation for between-aliquot measurements is larger.

POWER MEASUREMENT

Each sample was calorimetered twice: once in Calorimeter 111 and once in Calorimeter 133. The average difference between the two measurements for the 15 samples is -0.00016 W with a standard deviation of ± 0.0014 W. The average wattages have been used with uncertainties determined from heat standards: 0.5% for less than 0.1 W, 0.25% for 0.1-0.2 W, and 0.1% for greater than 0.2 W. The procedures of ANSI-N15.22 [7] were used to convert the isotopic abundances and power measurements to grams of plutonium.

RESULTS

The calorimetric assay data are summarized in Tables 2 and 3. Table 2 contains all 15 samples grouped by the uncertainties in their isotopic compositions. Groups I and II are not recommended as reference standards because the uncertainties in the plutonium isotopics cause large uncertainties in the plutonium weight when decaying these samples. Group III contains eight samples covering the weight range from 4.3 to 141.2 g of plutonium. These samples are recommended as reference standards and Table 3 shows the sample power and plutonium values through January 1, 1979. If the samples are to be used after that date, they should be reverified. (J. F. Lemming and J. Y. Jarvis)

Table 2 - EVALUATION RESULTS FOR PROPOSED STANDARDS

<u>Group</u>	<u>Sample</u>	<u>Calorimetry Date</u>	<u>Power (W)</u>	<u>Plutonium (g)</u>
I	AP	7/16/76	0.31237	108.7 + 8.3
	901	7/14/76	0.12686	42.8 + 1.4
	914	7/14/76	0.25258	84.1 + 2.0
	930	6/23/76	0.44796	141.9 + 2.8
II	712	7/6/76	0.01931	6.7 + 0.1
	858	7/9/76	0.01322	4.4 + 0.1
	978	7/6/76	0.37330	156.4 + 2.0
III	247	7/16/76	0.26846	93.2 + 1.3
	655	7/9/76	0.01655	4.3 + 0.1
	888	7/5/76	0.45278	141.2 + 2.7
	889	7/14/76	0.16147	50.1 + 1.0
	906	7/14/76	0.05352	13.9 + 0.3
	931	6/23/76	0.18241	47.2 + 1.1
	945	7/14/76	0.04425	11.4 + 0.3
	961	7/5/76	0.21034	87.0 + 0.5

Table 3 - EXTRAPOLATED RESULTS FOR RECOMMENDED STANDARDS (GROUP III)

Sample	Date	Power (W)	Plutonium (g)
247	7/16/76	0.26846	93.2 $\pm$ 1.3
	1/1/77	0.26994	93.2
	7/1/77	0.27149	93.1
	1/1/78	0.27302	93.1
	7/1/78	0.27448	93.1
	1/1/79	0.27594	93.1
655	7/9/76	0.01655	4.3 $\pm$ 0.1
	1/1/77	0.01671	4.3
	7/1/77	0.01686	4.3
	1/1/78	0.01702	4.3
	7/1/78	0.01716	4.3
	1/1/79	0.01731	4.3
888	7/5/76	0.45278	141.2 $\pm$ 2.7
	1/1/77	0.45629	141.1
	7/1/77	0.45973	141.1
	1/1/78	0.46314	141.1
	7/1/78	0.46640	141.0
	1/1/79	0.46964	141.0
889	7/14/76	0.16147	50.1 $\pm$ 1.0
	1/1/77	0.16266	50.1
	7/1/77	0.16388	50.1
	1/1/78	0.16510	50.1
	7/1/78	0.16626	50.1
	1/1/79	0.16741	50.0
906	7/14/76	0.05352	13.9 $\pm$ 0.3
	1/1/77	0.05402	13.9
	7/1/77	0.05454	13.8
	1/1/78	0.05506	13.8
	7/1/78	0.05555	13.8
	1/1/79	0.05604	13.8
931	6/23/76	0.18241	47.2 $\pm$ 1.1
	1/1/77	0.18430	47.2
	7/1/77	0.18604	47.2
	1/1/78	0.18776	47.2
	7/1/78	0.18941	47.2
	1/1/79	0.19104	47.1
945	7/14/76	0.04425	11.4 $\pm$ 0.3
	1/1/77	0.04466	11.4
	7/1/77	0.04508	11.4
	1/1/78	0.04550	11.4
	7/1/78	0.04590	11.4
	1/1/79	0.04632	11.4
961	7/5/76	0.21034	87.0 $\pm$ 0.5
	1/1/77	0.21093	87.0
	7/1/77	0.21151	87.0
	1/1/78	0.21208	87.0
	7/1/78	0.21262	86.9
	1/1/79	0.21317	86.9

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